

PATENT APPLICATION**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re application of

Docket No: Q82625

Tsukasa AGA

Appln. No.: 10/502,014

Group Art Unit: 1796

Confirmation No.: 8458

Examiner: William K. Cheung

Filed: July 20, 2004

For: AQUOES WATER- AND OIL-REPELLENT DISPERSION

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Takashi Enomoto, hereby declare and state:

THAT I am a citizen of Japan;

THAT I have received the master degree (department of chemical for materials) in 1986 from Mie University;

THAT I have been employed by DAIKIN INDUSTRIES LTD. since April 1, 1986, where I hold a position as researcher, with responsibility for research works on the development of the synthesis of fluorine-containing compound and the development of water and oil-repellent; and I worked in production department of fluorine-containing products between 1996 and 1999. Again, I started work for developing water and oil-repellent from 1999 to now. Especially, I worked in USA (DAIKIN AMERICA) between 2000 and 2006.

THAT I am familiar with the Office Action dated June 12, 2008; and

THAT the following experiments were carried out under my direct supervision.

EXPERIMENTATION

The following experiments were performed.

Properties are determined as follows:

Water- and oil-repellency

The polymer dispersion liquid is diluted with water to give a treatment liquid having a solid content of 0.08 % by weight. A polyester fabric is immersed in the treatment liquid, squeezed with a mangle to give a wet pickup of 65%, dried at 100°C for two minutes, heated at 160°C for one minute, and then subjected to an evaluation of water- and oil-repellency.

The water-repellency is expressed by the water repellency No. (cf. the following Table 1) determined by the spray method according to JIS (Japanese Industrial Standard) L-1092.

The oil-repellency is determined by dropping several drops of a test solution shown in the following Table 2 according to AATCC-TM118 on two positions of a surface of a test cloth and observing the penetration state of the drops after 30 seconds. The maximum point at which the test solution exhibits no penetration is expressed by the oil-repellency.

Table 1

| Water repellency No. | State |
|----------------------|----------------------------|
| 5 | No wet on the surface |
| 4 | Slight wet on the surface |
| 3 | Partial wet on the surface |
| 2 | Wet on the surface |
| 1 | Wet over the whole surface |

Table 2

| Oil-repel-lency | Test solution | Surface tension (dyne/cm, 25°C) |
|-----------------|---|---------------------------------|
| 8 | n-Heptane | 20.0 |
| 7 | n-Octane | 21.8 |
| 6 | n-Decane | 23.5 |
| 5 | n-Dodecane | 25.0 |
| 4 | n-Tetradecane | 26.7 |
| 3 | n-Hexadecane | 27.3 |
| 2 | n-Hexadecane/Nujol mixture solution (35/65 by weight) | 29.6 |
| 1 | Nujol | 31.2 |
| 0 | Inferior to 1 | - |

Mechanical stability

The aqueous dispersion is diluted with tap water to give a solid content of 0.2 % by weight. The diluted dispersion is stirred in a homomixer at 3,000 rpm for 10 minutes and generated scum is filtered with a black cotton fabric.

- : No scum generated
- △: Slight scum generated
- ×: Much scum generated

Chemical stability

The aqueous dispersion is diluted with tap water to give a solid content of 0.6 % by weight. 0.03 % by weight of a fixing agent for nylon is added to the diluted dispersion and intimately stirred. The generation of agglomerate is observed.

- : No agglomerate generated
- △: Slight agglomerate generated
- ×: Much agglomerate generated

Storage stability

After the aqueous dispersion (solid content: 30 % by weight) is stored at 40°C for one month, the generation of precipitate is observed.

○: No precipitation

△: Slight precipitation

×: Much precipitation

Following Experiment 1 is the same as Example 1 described in the present specification.

Experiment 1:

100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (n is 6, 8, 10, 12 and 14 (average of n is 8)) (FA), 45 g of stearyl acrylate (StA), 5 g of N-methylol acrylamide (N-MAM), 3 g of trimethyloctadecyl ammonium chloride (TMOAC), 12 g of polyoxypropylene polyoxyethylene isotridecyl ether (POPPOE-ITDE), 40 g of tripropylene glycol (TPG) and 200 g of pure water were stirred at 60°C for 15 minutes.

Then, with maintaining the temperature at 50°C to 60°C, the mixture was emulsified by a high pressure emulsifier manufactured by Gaulin, Inc. (throughput: 0.4 L/min.) at 400 kg/cm² for 10 minutes.

The mixture was transferred to a 1 L autoclave, the atmosphere of the autoclave was replaced with nitrogen and 0.75 g of V-50 was added to the mixture. The polymerization was performed with stirring at 60°C for 5 hours to give an aqueous water- and oil-repellent dispersion.

Used substances are shown in Table I.

The water- and oil-repellency, the mechanical stability, the chemical stability and storage stability of the aqueous water- and oil-repellent dispersion were evaluated. The results are shown in Table II.

Comparative Experiment 1

The same procedure as in Experiment 1 was repeated except that Polyoxypropylene polyethylene isopentadecyl ether (IPDE) $[(C_8H_{17})(C_6H_{13})CH-O[CH_2CH(CH_3)O]_x-(CH_2CH_2O)_yH]$, wherein x is 5 and y is 13.) was used instead of POPPOE-ITDE.

The results are shown in Table II.

TABLE I

| | Copolymer | Cationic surfactant | Nonionic surfactant | Organic solvent |
|--------------|--------------------------|---------------------|---------------------|-----------------|
| Experiment 1 | FA/StA/N-MAM=100/45/5(g) | TMOAC 3g | POPPOE-ITDE 12g | TPG 40g |
| Experiment 1 | FA/StA/N-MAM=100/45/5(g) | TMOAC 3g | POPPOE-IPDE 12g | TPG 40g |

The abbreviations shown in Experiments indicate the following compounds.

FA: $C_n F_{2n+1} CH_2 CH_2 OCOCH=CH_2$ ($n = 6, 8, 10, 12, 14$; Average is 8)

StA: Stearyl acrylate

N-MAM: N-Methylol acrylamide

TMOAC: Trimethyl octadecyl ammonium chloride

POPPOE-ITDE: Polyoxypropylene polyoxyethylene isotridecyl ether

POPPOE-IPDE: Polyoxypropylene polyoxyethylene isopentadecyl ether

TPG: Tripropylene glycol

TABLE II

| | Water- and oil-repency | | | | | | Mechanical stability | Chemical stability | Storage stability |
|--------------------------|------------------------|-------------|---------------|-------------|---------------|-------------|----------------------|--------------------|-------------------|
| | Initial | | Durability | | | | | | |
| | | | HL-0 | | DC-3 | | | | |
| | Water repency | Oil repency | Water repency | Oil repency | Water repency | Oil repency | | | |
| | Experiment 1 | 5 | 6 | 4 | 4 | 4 | | | |
| Comparative Experiment 1 | 5 | 6 | 4 | 4 | 3 | 3 | Δ | O | |

Evaluation method of water- and oil-repency

HL-0: Initial (before washing and cleaning)

HL-3: After 3 times washing according to a JIS L-0217-103 method

DC-3: After 3 times dry cleaning according to a JIS L-1092-322 method

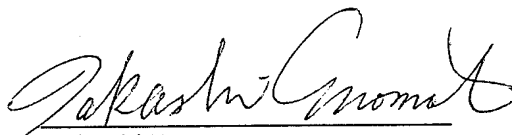
Water repency is according to a spray method of JIS L-1092, and

Oil repency is according to AATCC-118.

From the results, it is understood that polyoxypropylene polyoxyethylene isotridecyl ether gives better mechanical stability and chemical stability than polyoxypropylene polyoxyethylene isopentadecyl ether which is disclosed at column 10, lines 5-6 of Oharu et al. (US 6,610,775). Polyoxypropylene polyoxyethylene isotridecyl ether is included in the nonionic surfactant of the formula (I) recited in the present claim 1, but polyoxypropylene polyoxyethylene isopentadecyl ether is outside of the nonionic surfactant of the formula (I). The isotridecyl group has at least three side chains, while isopentadecyl group has only one side chain.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: Sep. 4, 2008


Takashi Enomoto